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Multi-Media, Multi-Concentration, Inorganic Analytical Service for Superfund (ILM04.1)*

Office of Emergency and Remedial Response
Analytical Operations/Data Quality Center (5204G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to EPA at such sites are a series of inorganic analytes and cyanide that are analyzed using inductively coupled plasma (ICP), atomic absorption (AA), and colorimetric techniques. The Analytical Operations/Data Quality Center (AOC) of the Office of Emergency and Remedial Response (OERR) offers an analytical service that provides data from the analysis of water and soil/sediment samples for inorganic analytes for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the inorganic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

The inorganic analytical service provides a technical and contractual framework for laboratories to utilize EPA/CLP analytical methods. These methods are used in the preparation, detection, and quantitative measurement of cyanide and 23 inorganic target analytes in both water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including quality assurance, quality control, and standard operating procedures, by which EPA evaluates the data. This service uses ICP, AA, and colorimetric methods to analyze the inorganic target analytes and cyanide. Three data delivery turnarounds are available to CLP customers: 7, 14, and 21-day turnaround after receipt of the last sample in the set. In addition, a 72-hour preliminary data submission option also is available for all turnaround times.

DATA USES

This analytical service provides data that EPA uses for a variety of purposes. Examples include determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of a hazardous waste site including site inspections, Hazard Ranking System scoring, remedial investigations/feasibility studies, remedial design, treatability studies, and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

TARGET ANALYTES

The analytes and detection limits for which this service is applicable are listed in **Table 1**. The list of target analytes for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since the inception of the CLP, analytes have been added to and deleted from the Target Analyte List, based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program. Specific detection limits are highly matrix dependent.

* ILM04.1 is an interim inorganic analytical service. Further changes are expected to be released under ILM05.0 for competition during calendar year 2000.

Table 1.
Target Analyte List and Contract Required Detection Limits (CRDLs) (ILM04.1)

Abbreviation	Analyte	CRDL[1] (µ/L)
Al	Aluminum	200
Sb	Antimony	60
As	Arsenic	10
Ba	Barium	200
Be	Beryllium	5
Cd	Cadmium	5
Ca	Calcium	5000
Cr	Chromium	10
Co	Cobalt	50
Cu	Copper	25
Fe	Iron	100
Pb	Lead	3
Mg	Magnesium	5000
Mn	Manganese	15
Hg	Mercury	0.2
Ni	Nickel	40
K	Potassium	5000
Se	Selenium	5
Ag	Silver	10
Na	Sodium	5000
Tl	Thallium	10
V	Vanadium	50
Zn	Zinc	20
Cn	Cyanide	10

Sample concentration exceeding five times the detection limit of the instrument or method in use may be reported even though the instrument or method detection limit is greater than the CRDL. This is illustrated in the following example:

For lead:
Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample Concentration = 220
CRDL = 3

[1]The CRDL is the instrument detection limit obtained in pure water.

METHODS AND INSTRUMENTATION

When storing samples, the use of a cooler temperature indicator bottle and the cooler temperature must be reported on Form DC-1 and in the Sample Delivery Group (SDG) Narrative. When applying AA methods, the Contractor may analyze the sample at a dilution so long as the raw concentration or absorbence of the diluted sample falls within the upper half of the calibration range. For ICP, an undiluted analysis of the sample is required.

If an insufficient sample amount (less than 90% of the required amount) is received to perform the analyses, the Contractor must contact the Sample Management Office (SMO) to report the problem. The same is required for multi-phase samples (e.g., two-phase liquid sample and oily sludge/sandy soil sample).

Table 2 summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include both hardcopy/ electronic data reporting forms and supporting raw data. The laboratory must submit data to EPA within 7, 14, 21-days, or preliminary data must be submitted within 72 hours after laboratory receipt of each sample in the set. EPA then processes the data through an automated Data Assessment Tool (DAT). DAT is a complete CLP data assessment package. DAT incorporates Contract Compliance Screening (CCS) and Computer-Aided Data Review and Evaluation (CADRE) to provide EPA Regions with PC-compatible reports, spreadsheets, and electronic files. These files can be provided to the Regions within 24 to 48 hours from the receipt of the data and can be used as a tool during the data validation process at the Region. This automated tool facilitates the transfer of analytical data into Regional databases. In addition to the Regional electronic reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has 4 days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

QUALITY ASSURANCE

The quality assurance (QA) process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of the quality required.

Table 2. Methods and Instruments

Analyte	Instrument	Method
Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn	Inductively Coupled Plasma (ICP)	Acid digestion followed by ICP analysis
As, Pb, Tl, Se	Graphite Furnace Atomic Absorption (GFAA)	Acid digestion followed by GFAA analysis
Ca, Mg, Na, K	Flame Atomic Absorption (FAA)	Acid digestion followed by FAA analysis
Hg	Cold Vapor Atomic Absorption (CVAA)	Acid and permanganate oxidation followed by CVAA analysis
CN	Manual and Semi-automated Colorimetric	Distillation followed by colorimetric analysis

Table 3. Quality Control

QC Operation	Frequency
Instrument Calibration	Daily or each time instrument is set up
Initial Calibration Verification	Following each instrument calibration
Initial Calibration Blank	Following each instrument calibration
Continuing Calibration Verification	Every 10 analytical samples or every 2 hours during a run and at the beginning and end of each run
Continuing Calibration Blank	Every 10 analytical samples or every 2 hours during a run and at the beginning and end of each run
Interference Check Sample	Every 20 analytical samples and at the beginning and end of each run
CRDL Standard for ICP	Every 20 analytical samples and at the beginning and end of each run
CRDL Standard for AA	At the beginning of each AA analytical run
Serial Dilution for ICP	For each matrix type and concentration level for each SDG
Preparation Blank	For each sample preparation, analysis, and matrix per batch of prepared samples
Laboratory Control Sample	For each sample preparation and analysis procedure for each batch
Matrix Spike Sample Analysis	For each matrix type, concentration level, and method for each SDG
Duplicate Sample Analysis	For each matrix type, concentration level, and method for each SDG
Post Digestion Spike	Each time matrix spike recovery is outside QC limits
Analytical Spike	For each analytical sample analyzed by furnace AA
Method of Standard Addition	When the analytical spike recovery is outside QC limits
Instrument Detection Limit Determination	Quarterly
Interelement Corrections	Annually for ICP instruments only
Linear Range Analysis	Quarterly for ICP instruments only

During the data collection effort, QA activities ensure that the quality control (QC) system is functioning effectively and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions. Each contract laboratory prepares a quality assurance plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, functional guidelines, and QA/ QC activities designed to achieve the data quality requirements for this analytical service.

QUALITY CONTROL

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analytical service are shown in **Table 3**.

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by AOC and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, and evaluates laboratory performance through the use of blind performance evaluation samples.

For more information, or for suggestions to improve this analytical service, please contact:

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